JOURNAL OF NANO- AND ELECTRONIC PHYSICS
Vol. **8** No 3, 03054(3pp) (2016)

ЖУРНАЛ НАНО- ТА ЕЛЕКТРОННОЇ ФІЗИКИ
Том **8** № 3, 03054(3cc) (2016)

Short Communication

The Micro and Nano- defects Formation during Czochralski Growth

V.G. Kosushkin[1], L.V. Kozhitov[2] I.A. Kaplunov[3]

[1] *Bauman Moscow State Technical University (Branch in Kaluga), 2, Bazhenov st., 248000 Kaluga, Russia*
[2] *National University of Science and Technology MISIS, 4, Leninskii pr., 119049 Moscow, Russia*
[3] *Tver State University*

(Received 04 May 2016; published online 03 October 2016)

The main idea was to determine the conditions of macro- and micro-defects formation in single crystals of large diameter grown by the Czochralski method. It was found that the value of the average density of dislocations in the crystal cannot be regarded as a definite criterion of origin or absence of the substructure. It was consider that the small-angle boundaries are the result polygonisation occurring mechanism slip or climb of dislocations in the crystal growth zone, closely related to the crystallization front. These boundaries can reach the solidification front, after which they inherited a growing crystal, forming macroblocks oriented along the growth axis.

Keywords: Single crystal, Structural defects, Poligonisation in dislocation structure.

DOI: 10.21272/jnep.8(3).03054 PACS numbers: 61.72. – y, 61.72.Ff

1. OBJECTIVES OF THE WORK

The main idea of our investigation was to determine the conditions of macro- and micro-defects formation in single crystals of large diameter grown by the Czochralski method.

We propose a technique which allows to implement the algorithm development technology based on a combination of theoretical and experimental approach on the example of the choice of parameters of single crystals of germanium with a diameter of 150 mm (100) orientation with an average dislocation density of less than 10^3 cm^{-2}, the absence of low-angle boundaries and heterogeneity of the impurity distribution over the cross section less than 5 % of the crystal.

The factors determining the conditions for obtaining single crystals with the given parameters in a Czochralski method, are known and described many times [1]. However, the number of control actions (ingot speed, travel speed of the crucible and the melt, the crystal and crucible rotation in the melt) must have defined values for each of the steps of growing ingot – the moment of separation before the seeding of the ingot from the melt in the crucible. Choosing a set of parameters and control actions should be preceded by a meaningful analysis of the reasons for the formation of unwanted defects in the crystal growth process.

The analysis of the characteristics the dislocation structure formation in germanium single crystals with a diameter of 150 mm, dislocation density of less than $1{\cdot}10^3$ cm^2 will be considered as an example.

The modern concepts of defect formation during the growth of single crystals shows that the dislocation structure formed under the influence of technological factors together and basically can be described in terms of the theory of thermoelastic stresses. General regularities of the formation of the dislocation structure of single crystals grown by the Czochralski method [2] can be applied to crystals of any diameter. However, as shown by our experiments, a particularly important role to play large-sized ingots processes of restructuring of the dislocation structure in the solid phase has formed crystal, leading to the formation of structural defects such as low angle boundaries. Investigation of this problem was the main focus in the work, as still get the homogeneous, low dislocations single crystals of germanium (100) orientation is difficult. In this paper we have used as empirical data, and well-known theory of dislocation motion and standard metallographic methods for determine this parameter.

2. EXPERIMENTAL PROCEDURES

Germanium single crystals were grown on standard crystals growth technique by the Czochralski method "Astra" [1]. Thermal unit included a system of three heaters – heating for crucible and two heaters for the growing crystal. This system made it possible to provide heat for the sustainable growth of monocrystalline and close to isothermal conditions cooling bars.

The standard etching procedure and is determined by standard distribution and average dislocation density of the sectional part was determine. The focus of research has been devoted to the problem of formation of low-angle boundaries in growing single crystals. From previous work [2-4] it is known that there is currently no single point of view on the mechanism of formation of low-angle boundaries during crystal growth. This is due to the presence of mechanisms, due to the nature of the material and the specific growing conditions. However, in the case of sufficiently pure crystals (when you can neglect the effects associated with the concentration supercooling at the growth front) defining role for the movement of dislocations in the field of thermal stresses. It is believed that the subboundaries are formed by redistribution formed under stresses dislocations. There are at least three mechanisms substructure formation are known [1]:

1. Polygonization slip as a result of the elastic interaction of dislocations of the same sign in the parallel

slip planes.

2. The formation of cells as a result of cross-slip. This process requires a thermal activation energy which decreases with increase in effective stress. Therefore, it appears at sufficiently high temperatures and voltages. Furthermore, the formation of a cellular dislocation structure on this mechanism depends greatly on the degree of splitting of dislocations. Furthermore, the formation of a cellular dislocation structure on this mechanism depends greatly on the degree of splitting of dislocations.

3. Polygonization "climb of" dislocation. It is possible only at high temperatures and the absence of split dislocations.

Dislocation walls resulting from polygonisation slip and climb have similar type. The only difference between them is that the distance between dislocations along the wall, formed by sliding can vary. When has one slip system, formed a rough polygonal substructure consisting of extended family parallel sub-boundaries perpendicular to the sliding direction. In the presence of several active slip systems are formed thinner substructure. Nearby dislocation walls can "grow together" as a result of the diffusion of dislocation to form one LABs with twice the density of dislocations. This mechanism explains the observed number of cases in the rebuilding of fine cellular substructure in rougher with long dense sub-boundaries.

To study the defects have been used by optical and scanning electron microscopy (SEM). To examine the surface topography of the sample by SEM secondary electron mode was used (SEI), the study was carried out on a scanning electron microscope JEOL JSM-6610LV. Analysis of the grown single crystals showed that the growth of single crystals with dislocation density over ~ $5 \cdot 10^3$ cm^{-2} may be accompanied by the formation of low-angle boundaries in them (Fig. 1).

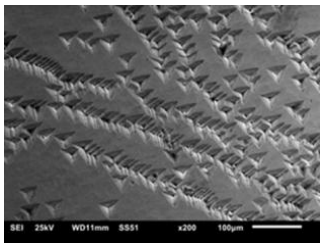

Fig. 1 – The images on a surface MUG Ge, grown by the Czochralski method in the [111] × 200 (c) (JEOL 6610 LV)

It is shown theoretically [1] that the relatively lower level of stress dislocation walls forming the cellular substructure, can occur at a lower average density of dislocations in the crystal. This is important for the case under consideration. This explains why in the semiconductor crystals in some cases with low thermal stress weakly expressed cellular dislocation structure is observed even in ingots with a relatively low dislocation density (less than $5 \cdot 10^3$ cm^{-2}). Physically, this is a consequence of dependence of the stability of the dislocation walls in relation to the current stress on the linear density of dislocations in the wall.

Thus, the value of the average density of dislocations in the crystal cannot be regarded as a definite criterion of origin or absence of the substructure. Indeed, for the formation of the expressed cellular distribution of dislocations, need appropriate kinetic conditions, determined by the temperature and the level of operating stresses. Therefore, for example, increasing of the growth rate does not lead to a noticeable change of the dislocation density or an increase in dopant concentration. This can reduce dislocation mobility and substantially decrease the tendency to form a cellular dislocation structure.

It can be assumed that the formation of blocks with minimal disorientation occurs through a mechanism of polygonisation. One of the most likely mechanisms controlling the formation of a cellular substructure, is a combination of cross slip of screw dislocations and climb. This is qualitatively consistent with a temperature range of education cellular substructure. This dependence of the activation energy of cross slip of stress can explain the distorting of slip bands and the formation of a cellular dislocation structure in the peripheral regions of the crystal, where there are maximum of thermoelastic stresses. Especially this trend is typical for crystals with large diameters. This mechanism supports a significant reduction in the probability of formation of sub-boundaries in doped semiconductor crystals. Dopants can lead to an increase in the width of the splitting of dislocations, making them difficult to climb and cross slip.

There are several points of view on the possible mechanisms of extended low-angle boundaries, forming macroblocks, elongated along the growth direction [4]. The most common are the following three:

1. It is assumed that the low-angle boundaries are formed by the closure of several sites disoriented growing layer that originated in different places of the solidification front.

2. Considers that the small-angle boundaries are the result polygonisation occurring mechanism slip or climb of dislocations in the crystal growth zone, closely related to the crystallization front.

3. The third possible mechanism is that in a high - distance dislocations moving. Dislocations move under the influence of thermal stresses in the part of the crystal in which the shear stresses are zero and form stable clusters of dislocations. In the particular case they extended low-angle boundaries.

For the true small-angle boundaries should be satisfied of Franc:

$$\vec{V} \times \vec{\alpha} = \sum \vec{b}_i (\vec{N}_i * \vec{V})$$

for any vector $\vec{V}$ in the plane of the boundary with the normal $\vec{n}$, where, $(\vec{N}_i = N_i (\vec{n} \times \vec{\xi}_i)$, $N_i = \left(\frac{1}{2}\right) d_i \cdot \sin\left(\frac{\theta}{2}\right)$, di – is the distance between dislocations; i – set in the border with the Burgers vector $\vec{b}_i$, $\vec{\xi}_i$ – the unit vector along the dislocation axis i – of the set; $\vec{\alpha}$ – the unit vector along the axis of rotation θ – rotation angle.

Of course, not all types are equally low-angle boundaries in real crystals grown from the melt. The occurrence of a certain limit with the growth depends on whether there is a dislocation in a crystal desired

types, as well as the possibility of the formation of the equilibrium concentrations in the boundary plane. This determined by the field of thermal stresses in the growing crystal and the crystallographic direction of growth.

The force $\vec{F}$ acting on a unit length of dislocation in the stress field $\vec{\sigma}$ is determined by the equation $\vec{F} = (\vec{b} \cdot \vec{\sigma}) \times \vec{\xi}$ with the component in the slip plane $F = [(\vec{b} \cdot \vec{\sigma}) \times \vec{\xi}][\vec{\xi} \times (\vec{b} \times \vec{\xi})] / (\vec{b} \times \vec{\xi})$.

This expression determines the magnitude and direction of the force that determines the motion of dislocations in the slip plane. In accordance with the mechanism of the formation of stable dislocation walls, dislocations, moving in their slip planes will be collected in areas where the condition $F_{sl} = 0$.

Knowing the tensor of thermal stress in the crystal $\hat{\sigma}$ and the possible types of dislocations is making possible to find the types and distribution of low-angle boundaries in the crystal. The calculations of thermal stress in the crystal with the experimentally determined types of dislocations, can determine that, in the radial heat sink, radial temperature gradients in the solid phase of the growing crystal must not exceed one - one and a half degrees per centimeter for "radial quench" available in the crystal dislocations and prevent redistribution in the slip line and low-angle boundaries.

Thermal system included three independently controllable heaters enabled the small temperature gradients in the solid phase during crystal growth a large length, which ultimately led to the problem of producing large-sized ingots of germanium (100) orientation with an average dislocation density less than 10^3 cm^{-2} without low-angle boundaries.

The analysis includes both theoretical consideration and experimental studies. It was found that the formation of low-angle boundaries in bulk germanium ingot grown by the Czochralski method is mainly due to the motion of dislocations in the solid phase crystal grown under the influence of radial temperature gradient. This allowed determining the temperature gradients in the melt and crystal growth providing sustained monocrystalline ingots.

Experiments were performed at the facility, equipped with a special device to measure and record temperature in the area melt during growth of germanium ingot diameter of 150 mm from the crucible of 300 mm diameter under a layer of flux – boron oxide thickness of 2 mm [1].

Preliminary experiments have shown that the change in rotational speed of the crucible (0-10 / min) or seeds (0-20 / min) had no significant effect on the nature of the layered heterogeneity.

Temperature gradients in the crystal, in which a single-crystal growth has been steady, is 20-25 deg/cm, in the column melt 12-22 deg/cm, melt away from the crystallization front 5-10 mm 5-8 deg/cm. The amplitude of the fluctuations of temperature was in the range of 2-5 degrees.

This work was supported by grant RFFI No 14-42-03046

REFERENCES

1. L.V. Kozhitov, V.G. Kosushkin, V.V. Krapuhin, Yu. Parkhomenko, *Materials Technology micro- and nanotechnology* (M.: MISA: 2007).
2. S.L. Kozhitov, Yu.N. Parchomenko, L.M. Chervyakov, L.V. Kozitov, *J. Nano- Electron. Phys.* **6** No 3, 03044 (2014).
3. S.L. Kozhitov, Yu.N. Parchomenko, L.M. Chervyakov, L.V. Kozitov, *J. Nano- Electron. Phys.* **6** No 3, 03043 (2014).
4. V.G. Kosushkin, L.V. Kozhitov, S.L. Kozhitov, *Proc. XI Int. Conf.* (FZGU, NITU MISA: Kursk: 2014).